Title: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) by SW846 Methods 8260C and 8260D

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 USEPA SW846 Methods 8260C and 8260D are used for the determination of volatile organic compounds in a variety of aqueous and solid matrices by purge and trap gas chromatography (GC)/mass spectrometery (MS). The methods are applicable to the compounds listed in Table 1 (below). Actual target compound lists are determined through regulatory or project specifications. Method performance criteria for each target analyte will be determined prior to sample analysis.

1.1.2 This SOP also describes the optional procedure for analyses of compounds using 8260C/8260D Selected Ion Monitoring (SIM). SIM analyses is specific to target compounds: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-Dioxane. Benzene and Chloroform if needed.

Table 1: Method Analytes

Compound	CAS #	Compound	CAS #
1,1,1,2-Tetrachloroethane	630-20-6	cis-1,2-Dichloroethene	156-59-2
1,1,1-Trichloroethane	71-55-6	cis-1,3-Dichloropropene	10061-01-5
1,1,1-Trifluoro-2,2-dichloroethane	306-83-2	Cyclohexane	110-82-7
1,1,2,2-Tetrachloroethane	79-34-5	Cyclopentene	142-29-0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Dibromomethane	74-95-3
1,1,2-Trichloroethane	79-00-5	Dichlorobromomethane	75-27-4
1,1-Dichloroethane	75-34-3	Dichlorodifluoromethane	75-71-8
1,1-Dichloroethene	75-35-4	Dichlorofluoromethane	75-43-4
1,1-Dichloropropene	563-58-6	Dimethylnaphthalene (total)	28804-88-8
1,1-Difluoroethane	75-37-6	Epichlorohydrin	106-89-8
1,2,3-Trichlorobenzene	87-61-6	Ethanol	64-17-5
1,2,3-Trichloropropane **(1)**	96-18-4	Ethyl acetate	141-78-6
1,2,3-Trimethylbenzene	526-73-8	Ethyl acrylate	140-88-5
1,2,4,5-Tetramethylbenzene	95-93-2	Ethyl ether	60-29-7
1,2,4-Trichlorobenzene	120-82-1	Ethyl methacrylate	97-63-2
1,2,4-Trimethylbenzene	95-63-6	Ethylbenzene	100-41-4
1,2-Dibromo-3-Chloropropane **(1)**	96-12-8	Ethylene Dibromide **(1)**	106-93-4
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	Hexachlorobutadiene	87-68-3
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	Hexane	110-54-3
1,2-Dichlorobenzene	95-50-1	Indan	496-11-7
1,2-Dichloroethane	107-06-2	Iodomethane	74-88-4
1,2-Dichloroethene, Total	540-59-0	Isobutyl alcohol	78-83-1
1,2-Dichloropropane	78-87-5	Isopropyl acetate	108-21-4
1,3,5-Trichlorobenzene	108-70-3	Isopropyl alcohol	67-63-0
1,3,5-Trimethylbenzene	108-67-8	Isopropyl ether	108-20-3
1,3-Dichlorobenzene	541-73-1	Isopropylbenzene	98-82-8
1,3-Dichloropropane	142-28-9	Methacrylonitrile	126-98-7
1,3-Dichloropropene, Total	542-75-6	Methyl acetate	79-20-9

Compound	CAS #	Compound	CAS #
1,4-Dichlorobenzene	106-46-7	Methyl acrylate	96-33-3
1,4-Dioxane **(1)**	123-91-1	Methyl methacrylate	80-62-6
1-Chloropropane	540-54-5	Methyl tert-butyl ether	1634-04-4
2,2,4-Trimethylpentane	540-84-1	Methylcyclohexane	108-87-2
2,2-Dichloropropane	594-20-7	Methylene Chloride	75-09-2
2,4,4-Trimethyl-1-pentene	107-39-1	Methylnaphthalene (total)	1321-94-4
2-Butanone (MEK)	78-93-3	Monochloropentafluoroethane	76-15-3
2-Chloro-1,3-butadiene	126-99-8	m-Xylene & p-Xylene	179601-23-1
2-Chloroethyl vinyl ether	110-75-8	Naphthalene	91-20-3
2-Chloropropane	75-29-6	n-Butanol	71-36-3
2-Chlorotoluene	95-49-8	n-Butyl acetate	123-86-4
2-Hexanone	591-78-6	n-Butyl acrylate	141-32-2
2-Methyl-1,3-butadiene	78-79-5	n-Butylbenzene	104-51-8
2-Methyl-2-propanol	75-65-0	n-Heptane	142-82-5
2-Nitropropane	79-46-9	n-Propyl acetate	109-60-4
2-Octanol	123-96-6	N-Propylbenzene	103-65-1
2-Octanone	111-13-7	o-Xylene	95-47-6
4-Chlorotoluene	106-43-4	p-Diethylbenzene	105-05-5
4-Ethyltoluene	622-96-8	Pentane	109-66-0
4-Isopropyltoluene	99-87-6	Propene	115-07-1
4-Methyl-2-pentanone (MIBK)	108-10-1	Propionitrile	107-12-0
Acetaldehyde	75-07-0	sec-Butylbenzene	135-98-8
Acetone	67-64-1	Styrene	100-42-5
Acetonitrile	75-05-8	Tert-amyl methyl ether	994-05-8
Acrolein	107-02-8	Tert-butyl ethyl ether	637-92-3
Acrylonitrile	107-13-1	tert-Butylbenzene	98-06-6
Allyl chloride	107-05-1	Tetrachloroethene	127-18-4
Amyl acetate (mixed isomers)	628-63-7	Tetrahydrofuran	109-99-9
Benzene **(1)**	71-43-2	Toluene	108-88-3
Benzyl chloride	100-44-7	Total BTEX	STL00431
Bromobenzene	108-86-1	trans-1,2-Dichloroethene	156-60-5
Bromoform	75-25-2	trans-1,3-Dichloropropene	10061-02-6
Bromomethane	74-83-9	trans-1,4-Dichloro-2-butene	110-57-6
Butadiene	106-99-0	Trichloroethene	79-01-6
Butyl Methacrylate	97-88-1	Trichlorofluoromethane	75-69-4
Camphene	79-92-5	Vinyl acetate	108-05-4
Camphor	76-22-2	Vinyl chloride	75-01-4
Carbon disulfide	75-15-0	Xylenes, Total	1330-20-7
Carbon tetrachloride	56-23-5	1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1
Chlorobenzene	108-90-7	1,4-Dioxane-d8 **(ISTD)**	17647-74-4
Chlorobromomethane	74-97-5	2-Butanone-d5 **(ISTD)**	24313-50-6
Chlorodibromomethane	124-48-1	Chlorobenzene-d5 **(ISTD)**	3114-55-4
Chlorodifluoromethane	75-45-6	Fluorobenzene **(ISTD)**	462-06-6
Chloroethane	75-00-3	TBA-d9 **(ISTD)**	25725-11-5
Chloroform **(1)**	67-66-3	1,2-Dichloroethane-d4 **(Surrogate)**	17060-07-0
Chloromethane	74-87-3	4-Bromofluorobenzene **(Surrogate)**	460-00-4

Compound	CAS #	Compound	CAS #
Chlorotrifluoroethene	79-38-9	Dibromofluoromethane **(Surrogate)**	1868-53-7
Chlorotrifluoromethane	75-72-9	Toluene-d8 **(Surrogate)**	2037-26-5

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).

1.1.3 Methods 8260C and 8260D can be used to quantitate most volatile organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. Water-soluble compounds can be included in this method, but quantitation limits will be higher due to poor purging efficiency.

1.1.4 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve (1 ug/l for most compounds). For a complete list of method detection limits (MDLs) and RLs, please see reference the current TALS (LIMS) active Method Limit Group database.

1.1.5 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 20 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

1.1.6 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 Methods 8260C and 8260D are used to determine volatile organic compounds in aqueous, non-aqueous and solid matrices. Sample preparation techniques vary, depending on the matrix and the level of contamination expected. Purge and trap techniques are used to introduce the sample to the GC/MS system. Refer to TestAmerica Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision and ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

2.2 All samples extracts are screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see

TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

2.3 An aliquot of sample containing internal standard and surrogate spiking solution is purged with nitrogen in a closed sparging vessel. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the volatiles onto a gas chromatograph column.

2.4 Analytes eluted from the capillary chromatograhy column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

2.5 For aqueous VOA samples submitted for New Jersey Groundwater Quality Standard (NJ GWQS) evaluation, a full scan analysis is initially performed using the 8260 methodology No further analysis by SIM is required if all of the following compounds are present above the full scan RL: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-dioxane, chloroform, vinyl chloride and benzene. If any of these compounds are undetected in the undiluted, full scan analysis, the sample must be analyzed via 8260C SIM or 8260D SIM for those compounds.

2.6 In order to meet lower reporting limits of 0.5ug/L for most analytes, 2.5 ug/L for ketones and generally lower limits for other non-routine analytical compounds, samples must be analyzed against an initial calibration with a low point at those levels. The corresponding TALS login method for low level aqueous analysis is 8260_LL. See Table 3b for initial calibration levels and spike amounts.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method is susceptible to contamination from a number of sources, including organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatiles analytical laboratory is housed in a separate building, away from the organic extraction lab area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatiles laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 Trip Blanks are shipped to clients with aqueous bottle ware as requested. The purpose of the trip blank is to detect and identify any VOC contamination of the samples while in transit to and from the lab. The blank is created at the laboratory by completely filling the volatile vial container with lab grade organic free deionized water and sealing the container Trip Blanks accompany bottle ware and samples through the sampling, storage and analysis stages as a check on contamination that may occur at these points.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.7 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.4 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.5 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551, 4100 Automatic Sampler/4660,4760 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4660,4760 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;

- Archon Autosampler/EST Encon concentrator.
- Archon/EST Evolution

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.3 An OI analytical purge trap #10 is used for the OI 4560,4660 and 4760 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: 30M long x 0.25mm ID, 1.4um film thickness, 20M x 0.18mm x 1um DB-624 and 20M long x 0.18 mm ID Restek Rtx-VMS capillary column with 1um film thickness or similar phase.

6.1.8 Mass Spectrometer (Agilent 5973/5975/5977): scanning from 35-260 amu every 0.9 seconds, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all EPA performance criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.1.9 GC/MS Interface: transfer lines heated to 180°C .

6.1.10 Data system: HP Chemstation II for data acquisition and TestAmerica Chrom for data processing.

6.2 Supplies

- Microsyringes: 10 ul to 1000 ul.
- Syringes: 5 ml to 25 ml gas-tight.
- Injection port liners: HP 18740-80200 or equivalent
- Volumetric flasks: Class “A” glassware, 5 ml to 500 ml.

- VOA vials: 20-ml and 40-ml glass with PTFE – faced septum.
- Vials: 2-ml amber glass with screw cap with Teflon-faced septa.
- Top loading analytical balance.
- Spatula: Narrow, stainless steel.
- Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.1.1) Reagent water is obtained from Millipore system. Other methods of preparing reagent water are acceptable, provided that the water produced meets method blank criteria.

7.1.2 Methanol: Ultra Resi-Analyzed, purge and trap grade, purchased from JT Baker or equivalent. (Cat # 9077-02)

7.1.2.1 Each lot of methanol is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 **Calibration Standards** Stock target compound analytical standard solutions are purchased mainly from Restek, Supelco, Inc, Absolute Standards and Spex although standards of similar quality from other suppliers may be substituted as required. Standards noted with an asterisk (*) are custom mixes made especially for TestAmerica Edison.

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 / Std #3 Gases*	2500 ppm	Restek	569722
8260 List 1 / Std #3 Gases – (SS)*	2500 ppm	Restek	569722 sec
8260 List 1 / Std #1 MegaMix*	1250-62500 ppm	Restek	569720
8260 List 1 / Std #1 MegaMix (SS)*	1250-62500 ppm	Restek	569720 sec
8260 List 1 / Std #2 Ketones *	12500 ppm	Restek	569721
8260 List 1 / Std #2 Ketones * (SS)	12500 ppm	Restek	569721 sec
8260 List 1 / Std #5 Acrolein *	20,000 ppm	Restek	568720
8260 List 1 / Std #5 Acrolein (SS)	20,000 ppm	Restek	568720 sec
8260 List 1 /Std #4 2 CEVE *	2500 ppm	Restek	569723
8260 List 1 /Std #4 2 CEVE (SS) *	2500 ppm	Restek	569723 sec

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 /Std #6 Vinyl Acetate *	5000 ppm	Restek	569724
8260 List 1 /Std #6 Vinyl Acetate (SS) *	5000 ppm	Restek	569724 sec
8260 List 2 / Std #1 Additions *	2500-62500ppm	Restek	568725
8260 List 2 / Std #1 Additions (SS) *	2500-62500 ppm	Restek	568725 sec
8260 List 3 / Std #1 Polar Additions *	2500-100000ppm	Restek	568728
8260 List 3 / Std #1 Polar Additions (SS) *	2500-100000 ppm	Restek	568728 sec
VOC Extra Standard 2015 *	2500-5000 ppm	Absolute	98593
VOC Extra Standard 2015 * (SS)	2500-5000 ppm	Absolute	98593
Epichlorohydrin	1000 ppm	Absolute	70377
Acrolein	5000 ppm	Restek	91980
Acrolein *	Neat	Sigma	110221
2-Freon Mix quote # 12258 *	2500ppm	Absolute	12258
2-Freon Mix quote # 12258 * (SS)	2500ppm	Absolute	12258
1,4-Dioxane	Neat	Sigma	360481
Epichlorohydrin	Neat	Sigma	45340
2-Chloroethylvinyl ether	Neat	Sigma	109983
1,4-Dioxane	1000 ppm	Absolute	70373
1,4-Dioxane	10000 ppm	Absolute	92785
Benzene	1000 ppm	Absolute	70025
Chloroform	1000 ppm	Absolute	70076

(1): The separate source for this material is not available as a distinct catalog number. Analyst must ensure that a separate lot of the material is selected and used as required.

An asterisk (*) indicates a custom standard mix.

7.2.1.1. Prepare stock solutions at volumes and concentrations indicated in Table 2 (Working Standards Preparation) by combining the indicated volumes of each stock solution into a volumetric flask corresponding to the total final volume. Dilute to the volume marker with methanol.

7.2.1.2. Prepare individual calibration standards as applicable per Section 9.2.2.1, Table 3, Initial Calibration Standards Preparation, Low Level Soil, Table 3a, Initial Calibration Standards Preparation (Low Level), Aqueous or Table 3B Initial Calibration Standards Preparation, Aqueous.

7.2.1.3. The 'Second Source' standards listed are used in the preparation of the Initial Calibration Verification (ICV) standard (see Tables 4 and 4a for ICV preparation instructions) and the Laboratory Control Standard (LCS) (see Section 9.1.3 and Tables 4 and 4a).

7.2.2 **Surrogate Standards:** Surrogate standard solutions are prepared from the stock solution (2500ppm)

Surrogate Standard Name	Concentration	Vendor	Catalog #
4-Bromofluorobenzene	2500ppm	Restek	567650
Toluene-d8			
1,2-Dichloroethane-d4			
Dibromofluoromethane			

7.2.2.1 A primary surrogate stock solution (2500 ppm each) is prepared from the neat standards as follows:

7.2.2.2 Secondary surrogate standard solutions are prepared at two (2) levels using the 2500 ppm primary stock solution as detailed in the table below:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume in MeOH/Total volume of MeOH
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	250ppm	10mL 9.0mL TV/M
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	50ppm	50mL 9.0mL TV/M

7.2.2.3 Methanol/Surrogate solution (2.5ug/mL): For methanol sampling field kits. Prepared by adding 1mL of 2500 ug/ml primary surrogate stock solution (see Section 7.2.2.1) to 1 L purge and trap grade methanol.

7.2.3 **Internal Standards:** Internal Standards Solutions are purchased from Restek:

Standard Name	Concentration	Vendor	Catalog #
8260 Internal Standard Mix: *Chlorobenzene-d5 *1,4-Dichlorobenzene-d4 *Fluorobenzene *1,4-Dioxane-d8 *TBA-d9	250-5000ppm	Restek	567649

7.2.4 **Internal Standard/Surrogate Mix (125 ppm each):** A solution containing both Internal Standards and Surrogates at 125 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500

ppm surrogate stock solution prepared in Section 7.2.2.1 and the 2500 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 20ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (125 ppm) For Aquatek Autosampler	2500 ppm Surrogate Mix	1.0ml	125 ppm each component
	250 Internal Std Mix	10 ml	

7.2.5 **Internal Standard/Surrogate Mix (SIM) (2.5/50 ppm each):** A solution containing both Internal Standards and Surrogates at 25 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 250 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 10ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (25 ppm) (SIM)	2500 ppm Surrogate Mix	10ul	2.5/50 ppm each component
	250 Internal Std Mix (Restek)	10ul	
1,4-Dioxane-d8	10000 ppm	50ul	

7.2.6 **GC/MS Instrument Performance Check (BFB):** The instrument performance check solution consists of 4-Bromofluorobenzene in addition to the other three surrogates in methanol. Prepare the solution at **50ppm as specified in section 7.2.2.2**. Assign an expiration date of 6 months.

7.2.7 All standards preparation information must be logged into the TALS Reagent Module. All pertinent information must be entered: Date prepared, Lot #'s, Expiration dates, Solvents used, Lab Lot # (expiration date), Manufacturer and Verification signature. Additionally, all prepped standards are typically given a unique Lot# and all information pertaining to standard preparation is entered into the GC/MS VOA Standard Preparation Log Book. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.8 Please refer to TestAmerica Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision. For Method 8260C and 8260D::

- Shelf Life of Standard:
  - Stock standards (Non-gases) - 6 months after opening vendor stock of up to 2000ppm, 3 years for 10,000ppm, 5 years for over 50,000ppm, or manufacturer's expiration date whichever comes first.
  - Stock Standards (Gases) - 2 months after opening vendor stock, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Non-gases) – 6 months after preparation, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Gases) – 1 week from the date of preparation for 50ppm and 2 weeks for 500ppm, or manufacturer's expiration date whichever comes first.
  - Daily Calibration Standards – 24 hours after preparation.

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- Storage Requirements:

  Aqueous standards are stored at 4°C and Methanol standards are stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass 40 ml vials	40 mLs	HCl, pH < 2; Cool 4 ^{o}C ± 2^{o}C	14 Days / preserved 7 Days / unpreserved	SW846 Method 5030
Waters	Glass 40 ml vials	40mLs	TSP,pH>11 Cool 4 ^{o}C ± 2^{o}C	14 Days / preserved	SW846 Method 5030

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils (Low)	Encore or Terracore (40 ml vials)	5 grams in 5 mls DI H_2O	Frozen Stored -7°C to -20°C	14 Days	SW846 Method 5035A
Soils (Med)	Encore or Terracore (40 ml vials)	5 grams in 10 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030
Soils (High)	Glass (Lab Prepared Kits)	10 grams in 25 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030

8.1.1 There are several methods of sampling soil. The recommended method is to take samples using an EnCore™ sampler or using a Terra Core™ sampling kit. At specific client request, unpreserved soil samples in 4oz jars may be accepted. For EnCore and Terra Core sampling, a separate jar is required for percent solids/moisture determination, unless one is supplied for another analysis.

8.1.2 For EnCore™ samplers, the 5g sample is extruded into a pre-weighed 40mL vial containing 5mL of methanol (medium level analysis) or reagent water (for low level, <50 µg/kg, analysis). The exact samples weight is determined as the difference between the vial + preservative weight and weight after the sample is added.

- Samples must be transferred (extruded from the sampler) and preserved within 48 hours of sampling.
- Water preserved samples are then frozen at <10°C. Methanol preserved samples may be stored at > 0.0 °C but < 6° °C or frozen.
- Methanol preserved samples are shaken for at least 2 minutes, and a portion of the methanol extract after settling may be transferred to a smaller Teflon-lined capped vial for storage below 6 °C
- Normally one (1) medium level and two (2) low-level samples are taken and preserved.
- One vial with a clean matrix of each preservation type is prepared at the same time as samples, to be used for LCS analysis. Spikes are not added until the time of analysis.
- Samples are spiked with internal standards and surrogates at the time of analysis.

8.1.3 Terra Core™ sampling kits are pre-preserved for use and immediate samples preservation in the field. Kits are shipped that include one (1) methanol preserved and two (2) reagent water preserved vials, along with a 4oz jar for solids/moisture analysis volume.

- Terra Core™ vials are immediately placed in the freezer (<-10°C) upon receipt at the lab. Methanol preserved vials are shaken for at least two (2) minutes to break up the solid and create the methanol extract.

- Terra Core™ vials are labeled with the weight of the vial and preservative. The vials are re-weighed prior to analysis to determine the weight of the solid sample added. It is important that labels NOT be added to these vials prior to weighing, because the weight of the label will add to the sample weight. Vials may be marked with indelible marker, or placed in a labeled, sectioned box until ID labels can be added after weighing.

8.2 Unpreserved soils - At client request, unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. A 5g portion of the sample is transferred to a 40mL vial and mixed with reagent water and/or methanol for analysis. Since this procedure is not compliant with SW5035A an NCM and case narrative statement describing the non-conformance must be included with any resulting data reported to the client.

8.3 Aqueous samples are stored in 40mL glass vials with Teflon lined septa at >0 and ≤ 6.0℃. Vials are required to have no headspace lar ger than a small pea.

8.3.1 Regulatory requirements for 2-Chloroethyl vinyl ether:

- 2-Chloroethyl vinyl ether: The stability of this compound is reduced when subjected to low pH, therefore samples for analysis to include 2-CEVE must be taken without acid preservation. Unpreserved samples must be analyzed within 7 days.

- SW846 Update V removed special preservation requirements for Acolein and Acylonitrile. These compounds may be analyzed for using a preserved sample vial.

8.4 Soil samples and water samples preserved to pH <2 with HCl have a maximum holding time is 14 days from sampling until the sample is analyzed. If water samples are known to be unpreserved, the holding time is 7 days from sampling to analysis.

8.4.1 Preserved water samples are checked to confirm the preservation pH AFTER analysis because the vials must not be opened prior to analysis. If the pH is found to be >2, this must be addressed in the case narrative.

8.5 Medium level solid methanol extracts, if taken at the time of preservation, are aliquoted into 4 mL glass vials with Teflon lined caps and stored at > 0.0 °C but ≤6.0℃ or frozen. The extracts are stored with minim um headspace.

8.6 Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After 1-2 weeks, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every samples	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method blanks** are analyzed every 12 hours immediately after successful calibration verification (ICV and CCV) and before any samples are analyzed during the 12 hour clock. Analyze the blank in the same manner as the associated samples.

9.1.1.1. Prepare an aqueous blank by filling a 40 mL vial with reagent water and placing it in the autosampler. The autosampler will add the internal standard and/or surrogate standard.

9.1.1.2. Prepare a medium or high level blank in a 50 mL volumetric flask by adding 1.0 mL of purge and trap grade methanol to reagent water and bringing up to volume with the reagent water. The appropriate volume of this mix is added to the purge vessel. The autosampler will automatically internal standard and/or surrogate standard.

9.1.1.3. Prepare a low- level soil blank in a 40 ml VOA vial by adding a magnetic stir bar and 5 ml of reagent water and placing the vial in the autosampler tray. An additional 5mL of reagent water plus 1uL of 250ppm Internal Standard/Surrogate Mix (see Section 7.2.4) will be added by the Archon prior to purging.

9.1.1.4. To be considered acceptable, the method blank must not have any target analytes above the reporting limit. For method 8260D method blank is acceptable when target analyte concentrations are less than one-half of the reporting limit. Method blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected or sample

concentrations/responses are >10x the blank If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Re-analysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed. Method blanks, trip blanks and other field blanks must be carried out through all stages of sample preparation and analysis.

9.1.1.5. Surrogate recoveries for the method blank must be within the laboratory generated limits. (Method 8260C /8260D requires the use of a minimum of three (3) surrogates. Since we are spiking with four (4) surrogates, either 1,2-Dichloroethane-d4 or dibromofluoromethane can be recovered outside of control limits without corrective action) .Internal standard area counts in the method blank must be within method specified limits. If any surrogate or internal standard is outside the limits, the method blank must re-analyzed.

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch which may contain up to 20 samples, and additional samples can be added to the batch for 14 days after the first sample was analyzed). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared (as described in Section 9.1.2.1) concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria which are updated annually. For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database.

9.1.2.1. Prepare the MS/MSD as follows:

9.1.2.1.1 Low Level Soil: The low level soil MS/MSD is prepared as detailed in the following table. This is prepared in duplicate (one for the MS, the other for the MSD) in a 5 ml syringe filled with reagent water. Once prepped the solution is added to separate 40 ml vials each containing 5 gram aliquots of the sample to be spiked :

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
Gas Mix Li	50ppm	2	20

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
8260 combined	50ppm	2	20
Acrolein	500 ppm	3	300
Propenes	50ppm (varied)	2	20 (varied)
Freons	50 ppm	2	20

9.1.2.1.2 **Aqueous Samples:** The MS/MSD for aqueous samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with an aliquot of sample to be spiked. Once prepped the solution is poured into a 40 ml VOA vial and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Sample	Final Concentration(ug/L)
Gas Mix Li	50ppm	20	20
8260 combined	50ppm	20	20
Acrolein	500 ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50	20	20

9.1.2.1.3 **Medium & High Level Soils:** The MS/MSD for medium/high level soils is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with reagent water which has been previously spiked with the methanol sample extract. Once prepped the solution is poured into a 40 ml VOA vial, the and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Reagent Water containing sample methanol extract	Final Concentration (ug/L)
Gas Mix Li	50ppm	20	20
8260 conbined	50ppm	20	20
Acrolein	500ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50 ppm	20	20

9.1.2.1.4 **SIM:** The MS/MSD for SIM samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 100 ml

volumetric flasks filled with an aliquot of sample to be spiked. Once prepped, two separate 10ml solution is poured into 40 ml VOA vials, 2ul of SIM IS/S is then added to each vial and loaded onto the purge and trap autosampler:

Standard Solution	Concentration	Volume of Standard Added to 100 ml of Sample (ul)	Final Concentration (ug/L)
8260SIM Mix1	10ppm	0.5	0.05
1,4-Dioxane	50ppm (varied)	10	5
Benzene/Chloroform	10ppm	0.5	0.05

9.1.2.2. An Laboratory Control Sample (LCS) /Laboratory Control Sample Duplicate (LCSD) may be substituted for the MS/MSD if insufficient sample volume is available (see Section 9.1.3).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be prepared analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (see For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference. If the LCS recovery results are outside the method specified, the LCS is reanalyzed. If, upon reanalysis, the LCS is it is still outside of limits the entire batch must be reanalyzed. For 8260D, when an LCS is prepared in the same manner as CCV, the same standard can be used as both the LCS and CCV.

9.1.3.1 For LCS preparation instructions please refer to Section 9.1.2.1 for low level soil introduction technique (note: use reagent water only, no solid matrix is used when preparing the LCS) and Sections 9.1.2.1.2 and 9.1.2.1.3 as applicable for aqueous/medium or high level solids introduction (note: use reagent water only, no sample or sample extract is used when preparing the LCS).

9.1.3.2 The LCS for SIM samples is prepared as detailed in the following table. This is prepared in a 200 ml volumetric flasks filled with organic free reagent water. Once prepped, 10ml of the solution is poured into a 40 ml VOA vial and 2ul IS/SS added manually and loaded onto the purge and trap autosampler

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 Mix1	10ppm	1	0.05
1,4-Dioxane	50ppm	20	5
Benzene/Chloroform	10ppm	1	0.05

9.1.3.3 A Laboratory Control Sample Duplicate (LCSD) is analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a four (4) component surrogate standard mix (see Section 7.2.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database).

9.1.4.1. Surrogate recovery limits are lab generated and are updated annually.

9.1.4.2. Surrogate recoveries are calculated for the blank, samples, and QC samples. Surrogate recovery is calculated as:

$$\frac{\text{Concentration found}}{\text{Concentration added}} \times 100 = \%\ \text{RECOVERY}$$

9.1.4.3. If the surrogate recoveries of any blank, sample, or QC sample fails to meet the current recovery criteria, the sample must be re-analyzed. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary. Methods 8260C and 8260D requires the use of a minimum of three (3) surrogates. As we spike with four (4) surrogates, one can be recovered outside of control limits without corrective action.

9.1.5. Internal Standards: All samples, blanks, standards and QC samples are spiked with a five (5) component internal standard mix (See Section 7.2.3). The response (area count) and retention time of each internal standard in all samples, standards, blanks and QC samples are monitored.

9.1.5.1. The internal standard responses must be within -50 +100% of its corresponding internal standard in the mid-level calibration standard or the active calibration curve. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.1.5.2. Internal standard retention time is evaluated immediately after acquisition. The retention times of the internal standards must be within ±30 seconds of the internal standards from the mid point standard of the initial calibration or the calibration verification standard. Any blank, sample, or QC sample that fails to meet these criteria must be re-analyzed.

9.2 Instrument QC

9.2.1 GC/MS Instrument Performance Check (BFB): The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection or purging of 50ng of 4-Bromofluorobenzene (BFB) meets the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all BFB key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. For method 8260D tune checks are only required prior to initial calibration. (**NOTE**: see Method Modifications in Section 16.0).

BFB Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent of the base peak
95	Base peak, 100% relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	Greater than 50% of the base peak
175	5.0-9.0 percent of mass 174
176	Greater than 95.0% but less than 101% of mass174
177	5.0-9.0 percent of mass 176

9.2.1.1. The BFB mass spectrum may be evaluated using one of the procedures listed below. The spectrum may be background subtracted using a single peak no more than 20 scans before the peak apex. The BFB spectrum must meet the technical acceptance criteria listed in the table above:

- A single scan on the peak;
- An average of the peak;
- Use of three scan averaging and background subtraction techniques. Select the scan at the BFB peak apex, add +1 scan from the apex and -1 scans from the apex;

9.2.1.2. BFB parameter settings are stored in a tune file, which ill be used in all subsequent analysis of standards and samples.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. **Initial Calibration:** The initial calibration range consists of a five-point concentrations (six points for second order regression) of analytical standards prepared as described in Tables 3, 3A and 3B as applicable (attached). The initial calibration range must be analyzed only after the BFB instrument performance check has met the criteria in Section 9.2.1. A separate initial calibration range is analyzed for each sample introduction technique. The last initial calibration standard may be used to be the start of the 12 hour clock for samples analyzed after initial calibration. Verify closely eluting isomers resolution in the mid-point concentration of the ICAL. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is less than or equal to 50% of the average of the two peak heights. This should also be checked in the daily CC's.

9.2.2.2. If analysis by the SIM technique is required, prepare calibration standards for, Vinyl Chloride, Chloroform, Benzene 1,2-dibromoethane, 1,2,3-Trichloropropane and 1,2-dibromo-3-chloropropane at concentrations of 0.02, 0.04, .05, 0.10, 0.20, 0.50, 1.0 and 2.0 ppb; 1,4-Dioxane at 0.4, 1, 5, 10, 20, 30, 40 and 50ppb. See Table 5 that summarizes the preparation information.

9.2.2.3. **Initial Calibration Verification (ICV):** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.1.3 and Tables 4 and 4a (full scan) and Table 6 (SIM) (attached). The ICV must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3 **Continuing Calibration Verification (CCV):** A approximately mid-point (20ug/ml and 0.050/5ug/ml for SIM) Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the BFB instrument performance check. BFB is not a requirement for 8260D CCV verification. The CCV is prepared as detailed in Section 7.2.1.1 and Table 3 (attached).

9.2.4 **Calibration Acceptance Summary**

9.2.4.1. **Retention Time:** The relative retention times of each compound in the five calibration standards must agree within 0.06 relative retention time units.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. After the initial calibration range has been analyzed as detailed in Section 10.3.3 the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion for the compound (see attached Table 7)
Ais = Area characteristic ion of internal standard (see attached Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound using the five or six RFs from the initial calibration range.

9.2.4.2.2. The average RFs of the target analytes listed in the table below must meet the indicated minimum RF criteria:

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone *	0.050
Carbon disulfide	0.100
Methyl Acetate *	0.005
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone *	0.050
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone *	0.050
Toluene	0.400

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone*	0.050
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Note: Alternate ions chosen for the analytes in the table above may result in lower than recommended value

* These values are lower than method recommended values.

9.2.4.2.3. Any individual analyte that fails the minimum response factor above must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV

9.2.4.2.4. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

The % RSD of the common target compounds listed above must be ≤20% in order for the calibration range to be acceptable. If more than 10% of the compounds exceed the 20%RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, appropriate instrument maintenance like source cleaning should be performed. Any compound that do not meet the 20%

RSD or 0.99 correlation coefficient criteria must be flagged as estimated for detects.

9.2.4.2.5. For all compounds (including those analyzed by SIM): in order to assume linearity, the % RSD of the RRF's for each target analyte must be ≤20%.

9.2.4.2.6. If the above listed criteria is met, the system can be assumed to be linear, sample analysis may begin and the average RF from the initial calibration range may be used to quantitate all samples.

9.2.4.2.7. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.6.1 Linear regression: Calculate the first order linear regression for any compound which did not meet the 20% RSD criteria. The r value (Correlation Coefficient) of the equation must be ≥0.99 for linear regression to be employed.

9.2.4.2.6.2 Quadratic (or second order) regression: may be used if the linear regression correlation coefficient exceeds criteria. Quadratic regression requires the use of a minimum six calibration points. If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be $\geq$ 0.99

9.2.4.2.8. If neither of the alternative calibration techniques meets acceptance criteria i.e for more than 10% of the analytes fail both 20%RSD and 0.990 the calibration is not valid. Corrective action must be taken and the initial calibration range reanalyzed.

9.2.4.2.9. Non-detect results for any analyte that fails both 20%RSD and 0.990 correlation coefficient may be reported without flagging if (and only if) there has been a successful analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative criteria in the method must be met. Flagging of detected analytes results as estimated is discouraged when the 20%RSD and 0.990 criteria fails. In general no more than one or two of the poorest performing analytes should fail both criteria.

9.2.4.2.10. Due to significant bias to the lower portion of a calibration curve using the linear regression fit model a quantitation check on the viability of the lowest calibration point should be performed by re-fitting the

response from the low concentration calibration standard back into the curve as if it were an unknown sample (rename the lower point calibration file as a separate data file before re-processing). The results should be within ±30% of the standard's true concentration. This is not required for average RF or quadratic fits. Additionally forcing a linear regression through zero will meet the requirement of not re-fitting. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered 'out of control'. Report those target analyte outliers as estimated when the concentration is at or near the lowest calibration point and/or report to the next reporting level (i.e., the next higher calibration point for the analyte).

9.2.4.2.11. For additional detail refer to TestAmerica Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, latest revision.

9.2.4.3. Calibration Point Read-back Criteria: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be < 50%. Relative standard error (RSE) can also be used and must be ≤ 20% for each calibration point. See section 11.10 for the Calculation of the %Error.

9.2.4.4. Initial Calibration Verification (ICV): Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 9.2.2.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed this criteria as long as their recoveries are within 65-135%. For the poor performers the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria

proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.5. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a BFB instrument performance check, and analysis of a calibration verification standard.

9.2.4.4.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of BFB. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For method 8260D, tune verification is not required for daily CCV.

9.2.4.4.1.1 Calibration Verification: Analyze the calibration verification standard immediately after a BFB that meets criteria. For method 8260D, BFB is not needed. Use the mid point calibration standard (20ug/L). **NOTE:** The same sample introduction technique employed for the initial six-point calibration must be used for the calibration verification.

9.2.4.4.1.2 Calculate response factors (RF) for each compound using the internal standard method.

9.2.4.4.1.3 The RFs must meet the minimum RF criteria listed in the table in Section 9.2.4.2.2.

9.2.4.4.1.4 Calculate the % Difference for each response factor in the calibration check standard vs. the response factors from the initial calibration.

9.2.4.4.1.5 If the percent difference/drift (%D) for the compounds listed in the table in Section 9.2.4.2.2 is ≤20%, the initial calibration is assumed to be valid. If the ≤20% D criteria is not met for more than 20% of the compounds

in the initial calibration, corrective action/ investigation may be taken. After corrective action, another calibration verification standard may be injected. If the response for the analyte is still not ≤20%, a new initial calibration range must be generated.

9.2.4.4.1.6 For the poor performing compounds listed below that fail the 20%D or 50%D criteria adequate sensitivity may be demonstrated by including a low level standard (LLCCV) in the analytical batch.

Poor Performers	
Acetone	Acrolein
Carbon disulfide	1,4-Dioxane
2-Butanone	Cyclohexane
2-Hexanone	Methyl cyclohexane
4-Methyl-2-pentanone	Benzyl chloride
Chlorodibromomethane	Naphthalene
1,2-Dibromo-3-chloropropane	Cis-Dichloropropene
Bromomethane	Trans-Dichloropropene
Chloroethane	All Alcohols

When samples have non-detects for an analyte that fails the SOP criteria with low recovery a low level CCV must be analyzed in the batch as a demonstration of adequate sensitivity. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. Any sample detects for an analyte that fails the SOP criteria must be flagged as estimated, or detailed in the case narrative. In all cases every effort should be made to re-analyze on an instrument with a passing CCV.

9.2.4.4.1.7 Percent drift is used instead of percent difference in calibrations employing either the linear or second order regression modes.

9.2.4.4.1.8 For the compounds not listed in the table in Section 9.2.4.2.2: No one individual compound of interest may exceed 50%D. For SIM analysis the %D is 20%.

9.2.4.4.1.9 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the

latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.4.1.10 Internal standard area response is also evaluated immediately after acquisition. The response (area count) of each internal standard in the calibration verification standard must be within 50% - 100% of its corresponding internal standard in the mid-level calibration standard of the initial calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The instrument operating parameters are set as follows at the beginning of a method of analysis and remain constant throughout the entire analytical procedure

10.1.1.1 Full Scan Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C

Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

10.1.1.2 SIM Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

SIM Parameters:

Group 1
Plot 1 Ion: 51.0/96

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	51.0	100	58.0	100	65.0	100
	67.0	100	70.0	100	88.0	100
	96.0	100	78.0	100	83.0	100
	85.0	100	62.0	100	64.0	100

Group 2
Group Start Time: 6.20
Plot 1 Ion: 82/117

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	82.0	100	107.0	100	109.0	100
	117.0	100				

Group 3
Group Start Time: 8.50
Plot 1 Ion: 75/157

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	75.0	100	95.0	100	150.0	100
	152.0	100	152.0	100	157.0	100
	174.0	100				

10.2. Sample Preparation

10.2.1. Screening: All samples extracts must be screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

10.2.2. Aqueous Samples: Unopened 40 mls vials with aqueous samples are placed in an Archon autosampler. 1 uL of Internal Standard/Surrogate Mix (see Section 7.2.4) is added by the Archon as the 5 mL of the sample passes through the sample loop.

10.2.3. Medium or high level soils: Medium or high level extracts that will be run on an Archon autosampler are prepared in 50mL volumetric flasks. The Archon can be set up to add 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) to each sample as the 5mL portion passes through the sample loop.

10.2.4. Low level soils: Low level soils must be run on an Archon autosampler. 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3

and 7.2.2.2) and 5mL reagent water is added to each sample vial by the Archon immediately before the sample is purged.

10.2.5. **SIM analysis:** Aliquot 10ml of sample and manually add 2ul of 2.5/50ppm of internal standard/surrogate mix. Load to soil section of the autosampler for heated purge.

10.3. Instrument Performance and Calibration Sequence

10.3.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.3.2. Analyze the Instrument Performance Check Standard (BFB) as discussed in Section 9.2.1.

10.3.3. A unique initial calibration is then prepared for each sample introduction technique.:

10.3.3.1 **40 ml VOA Vial (Aqueous/Medium-High Level Soils):** Prepare aqueous calibration standards at six concentration levels for each parameter by adding the volumes of working standards listed in Table 3 to a 50mL volumetric flask of reagent water. Pour the calibration standards into 40mL VOA vials and load into the autosampler tray. If the internal standard is to be added by the Archon/OI autosamplers the addition of internal standard into the 50ml volumetric flaks may be omitted.

10.3.3.2 **40 ml VOA Vial (Low Level Soils):** If the calibration is for low-level soils prepared according to Method 5035AA, the calibration standards must be prepared by adding the volumes of working standards listed in Table 3 into a 5 mL syringe filled with reagent water and pouring the prepared standards into 40 mL VOA vials containing a magnetic stir bar.

10.3.4. Purge the standard for 11 minutes.

10.3.5. After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C for VOCARB, 190°C for #10 and backflushing it with helium.

10.3.6. Begin the GC temperature program and data acquisition.

10.3.7. Re-condition the trap by baking for 12 minutes at 260°C for VOCARB, 210°C for #10.

10.3.8. Cool the trap to (<31°C). The trap is now ready for the next sample.

10.3.9. Transfer data to network, and process using CHROM software.

10.4. Sample Analysis Sequence

10.4.1. Once the initial calibration has been verified by successful analysis of an ICV and Method Blank, analysis of samples may begin.

10.4.2. Samples must be analyzed under the same instrument conditions and using the same injection volume as the calibration standards.

10.4.3. Equilibrate all samples to room temperature prior to analysis.

10.4.4. If the sample concentration exceeds that of the range, the sample must be diluted and re-analyzed.

10.4.5. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.5. Data Processing

10.5.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled in the Chrom system. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.5.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.5.3. Data is transferred from the acquisition PC to the network for auto-processing with CHROM software.

10.5.4. Each data file is checked for correct information including sample number, job number, QA batch, dilution factor, initial volume, final volume, and % moisture.

10.5.5. The data processing service from Chrom queries LIMS for the sample processing parameters.

10.5.6. Each data file is processed using calibration factors from the most recent initial calibration, quantitation from the daily calibration verification standard is not permitted.

10.5.7. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8260C and 8260D are listed in Table 7.

10.6. Interpretation and Qualitative Identification:

10.6.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.6.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.6.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.6.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.6.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.6.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.6.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.6.1.7. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Otherwise, structural isomers are identified as isomeric pairs.

10.6.1.8. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.6.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of

greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.6.2.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.6.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.6.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.6.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6.2.6 If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc..).

10.7. Data Reporting

10.7.1. Final Report. LIMS TALS system automatically produces a data report consisting of key, hardcopy reports corresponding to specific data reporting requirements.

10.7.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.7.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.7.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.7.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.7.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.7.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

11.0. Calculations / Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method.

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3.. See Section 9.2.4.2 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.1.4. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RRF)(Vs)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Ais	=	Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Vs = Volume of sample purged (ml)

11.1.5. Low Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)}{(Ais)(RRF)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Ws = Weight of sample purged (g)

11.1.6. Medium Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)(Vt)(1000)(D)}{(Ais)(RRF)(Va)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF	=	Average relative response factor from the initial calibration.
Va	=	Volume of the aliquot of sample methanol extract added to reagent water for purging in ul
Vt	=	Total volume of methanol extract in milliliters
Ws	=	Weight of sample purged (g)

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method . For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 7)
Ais = Area characteristic ion of associated internal standard (See Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.4. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.2. (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.5. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} X\ 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.6. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.7. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

11.8. Accuracy:

$$\text{ICV , CCV and LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.9. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.10. Calculation of Percent (%) Error:

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i [1] = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the TestAmerica corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The LLOQ shall be verified annually on each instrument used for client sample analysis.

12.3.4 Recovery of each analyte must meet the laboratory established LCS

recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0 Pollution Control

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision. The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples/Returned Methanol Preservative - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1. United States Environmental Protection Agency, “Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Wastes, SW846, August 2006.

15.2. .United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.3. United States Environmental Protection Agency, “Method SW8000D: Determinative Chromatographic Separations”, Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012.

15.4. U.S. EPA. 2003. "Method 5030C (SW-846): Purge-and-Trap for Aqueous Samples," Revision 3. Washington, DC.

15.5. U.S. EPA. 2002. "Method 5035A (SW-846): Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1. Washington, DC.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.7. TestAmerica Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.8. TestAmerica Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision.

15.9. TestAmerica Edison ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

15.10. TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

15.11. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.12. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.13. TestAmerica Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision

15.14. TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision

15.15. TestAmerica Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, current revision.

15.16. TestAmerica Edison SOP No. ED-GEN-022, *Training*, current revision.

15.17. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision

15.18. TestAmerica Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.19. TestAmerica Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.20. TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision

16.0 Method Modifications:

16.1 Method 8260D requires the BFB tune standard to be analyzed once prior to an ICAL and not daily after that prior to sample analysis. The laboratory will analyze the BFB tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8260B/8260C for tune evaluation, rather than the criteria suggested in Table 3 of Method 8260D.

17.0 Attachments

N/A

18.0 Revision History

- Revision 8, dated 07/16//2020

    - Updated throughout to include requirements of SW 8260D and 8000D.
    - Updated Table 1 with full current analyte list.
    - Add text to Section 1.1.6 detailing procedures for documenting method variations via NCMs.
    - Section 2.6: clarified text regrading lower than standard RLs.
    - Section 4.4: clarified text regarding trip blanks.
    - Section 7.1.1: revised source and details of organic free water.
    - Section 8.0: added handling and preservation details for various soil and aqueous sample types.
    - Section 9.1.1.4: Added that concentrations allowed in blanks (one half of RL), how blank concentration relates to sample concentration (<1/10) and some guidance on re-analysis when concentration exceeds criteria.
    - Section 9.1.3: added that CCV/LCS can be the same run.
    - Tune verifications as not required for daily CCV updated throughout.
    - Section 9.2.2.1.: Allowance for last calibration standard to be the start of 12-hour clock for samples analyzed after initial calibration. Calculations for verifying peak resolution
    - New Section 9.2.4.2 added: Calibration Point Read Back Criteria
    - Section 11.10: added formula for calculation of Percent Error.
    - Section 12 (Method Performance) updated to include new MDL procedure and annual LLOQ procedure.
    - Updated references in Section 15 as necessary.

- Revision 7, dated 04/01/2020:

    - Updated formatting and branding to Eurofins
    - Sec 7.2.8: Revised Expiration dates based on concentration level and corrected storage requirements.
    - Sec 8.1: Changed Storage Blanks storage period from 1 week to 1-2 weeks.

- Revision 6, dated 01/17/2018:

    - Revised Table 5 and 6: revised to add additional levels plus Benzene and chloroform and to updated concentration level of ICV to current level.

    - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

    - Section 7.2.5: SIM IS/SS mix corrected to reflect lower concentration of IS mix .

    - Section 9.1.2.1.4: SIM MS/MSD preparation revised.

    - Section 9.1.3.2: SIM LCS/LCSD preparation revised.

    - Section 9.2.2.2: additional SIM levels added.

- Section 9.2.3: SIM CCV level concentration revised to reflect lower concentration.
- Section 10.2.5: New SIM analysis preparation narrative added
- Section: 6.1: New instrumentation added.
- Section: Section: 9.1.4.3: Revised to have any one surrogate out without the need for corrective action. This corrects previous narrative of one surrogate out of two.

- Revision 5, dated 12/11//2015:
  - Revised Table 5: new concentration of low standard (1,4-dioxane only).
- Revision 4, dated 12/08//2014:
  - Section 9.2.4.2.2: Table revised to reflect minimum RF of 0.050 for following compounds: acetone, 2-butanone, 4-methyl-2-pentanone, 2-hexanone.
  - Section 9.2.4.3: added statement 'for poor performers the range is 50-150%'.
- Revision 3, dated 11/10/2014:
  - Tables 1 and 7: added 1,2,4,5-Trimethylbenzene, 1,4-Diethylbenzene, Butadiene, 1,4-Difluorobenzene, 1-Chlorohexane, Freon 114, Freon 123a, Isooctane, 4-Ethyltoluene, t-Amyl Alcohol, Chlorofluoroethylene to list of target compounds and list of standard sources.
  - Section 2.5: added chloroform, vinyl chloride and benzene to the list of SIM analytes addressed in this section.
  - Section 2.6: revised the concentration of the low ketone standard to 2.5 ug/l.
  - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items. All standards prep tables revised to reflect current standard prep instructions.
  - Section 8. Preservation by TSP and holding time is added.
  - Section 9.1.2.1: updated source of standards used in various spiking solutions.
  - Section 9.1.3: LCS/MS/MSD. Preparation tables now indicate using calibration mix and not the second source mix.
  - Sections 9.1.4.3 and 9.1.1 : Revised to indicate that we are now spiking with 4 surrogates instead of the method required 3. One surrogate is now allowed to be out of limit criteria for either 1,2-Dichloroethane-d4 and Dibromofluoromethane.
  - Section 9.2.2: Chloroform, Vinyl Chloride and Benzene added as SIM compounds.

- Section 9.2.4.2.3.1. A list of 'poor performing compounds' is added with a ICAL RSD criteria of 50%.

- Section 9.2.4.3: now specifies that up to 10% of the compounds are allowed to exceed the 70-130% ICV recovery criteria as long as their recoveries are within 65-135%..

- Section 9.2.4.4.1.6: Added the following to the first sentence: '…or 50%D for the poor performing compounds'.

- Section 10.1.1.2: updated masses/dwell time for Group 1 under SIM Parameters.

- Throughout document as appropriate: Replaced references to Target with references to CHROM

- Added Section 10.5.5: "The data processing service from Chrom queries LIMS for the sample processing parameters."

- Revision 2, dated 11/04/2013:

  - Tables 1 and 7: added methyl acrylate, 1-methylnaphthalene and 2-methylnapthalene.

- Revision 1, dated 09/16/2011:

  - Tables 1 and 7: added cyclopentene, 2-chloro-1,3-butadiene, methacrylonitrile, propionitrile, ethyl methacrylate, 2-nitropropane, indan and isobutyl alcohol to list of target compounds and list of standards sources.

  - Section 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

  - Table 3: Initial Calibration Standards Preparation: is now split into three tables to include aqueous low level analysis.

  - Table 5: added following footnote:

    Levels 1 and 2 respectively are prepared in 500ml and 100ml final volumes
    [1]This level is also used as the Continuing Calibration Verification.

- Revision 0, dated 02/15/2011: New

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
Gas Mix Hi	Gas (Hi)	Restek	567645	5ml mL	2000 ppm	500 ppm	20mL 15mL TV/M
Gas Mix Li	Gas (Li)	Restek	567645	500 uL	2000 ppm	50 ppm	20mL 19.5mL TV/M
8260Mix 1	Mix 1 (Hi)	Restek	567641 567646 567642 568022	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10ml
8260 combined	Mix 1 (Li)	Restek	567641 567646 567642 568022 567643 568018 568713 568722 568723	1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml	2000 ppm	50 ppm	40ml 31ml TV/M
Acrolein	AC	Restek	82402	1.0ml	20000 ppm	500 ppm	40ml 39ml TV/M
8260 Mix 2	Mix 2 (Hi)	Restek	567643 568722 568019-fl 568713-fl	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10mL
8260 Mix 3	Mix 3 (Hi)	Restek	568723 568021-fl	2.5ml 2.5ml	2000 ppm	500 ppm	10ml 5ml TV/M
1,4-Dioxane	1,4-Dioxane	Supelco	360481	483.6ul	Neat	50000 ppm	10ml/9.52TVM
1,4-Dioxane	1,4-Dioxane	Supelco	NA	100ul	50000 ppm	500 ppm	10ml/9.90TVM
Propenes*	Propenes	Supelco	21240202	NA	1000/2000 ppm	NA	NA
Propenes*	Propenes	Supelco	21240202	1ml	1000/2000 ppm	50 ppm (varied)	20ml/ 19ml
Gas SS	Gas SS	Restek	567645.sec	1ml	2000ppm	50 ppm	40ml 39ml/TV/M
8260 Mix 1 SIM	8260 Mix 1 SIM	Supelco	5-02111	50 ul	2000ppm	10 ppm	10ml 9.95 TV/M
1,4-Dioxane SIM	1,4-Dioxane	Supelco	NA	100 ul	50000 ppm	500 ppm	10ml/9.90TVM

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
8260 SS	8260 SS	Restek	567641.sec 567646.sec 567642.sec 568022- sl 567643.sec 568019- sl 568713- sl 568722.sec 568723.sec 568021- sl	1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml	2000 ppm	50 ppm	40 ml 30 ml TV/M
Acrolein SS	AC SS	Restek	568720.sec	1 ml	20000 ppm	500 ppm	40 ml 39 ml TV/M
Propenes SS	Propenes SS	Supelco		1 ml	1000/2000 ppm	50/100 ppm	40 ml 39 ml TV/M
8260Mix 1 SIM SS	SIM MIX1 SS	Supelco	5S-02111	50ul	2000 ppm	10 ppm	10ml 9.95 TV/M
Benzene/ Chloroform	Ben/chl	Absolute	70025/ 70076	100ul each	1000ppm	10ppm	10ml 9.90 TV/M
1,4-Dioxane (SS)	1,4-Dioxane	Absolute	70373	1ml	1000 ppm	500 ppm	2ml/1ml TV/M

Asterisk (*) indicates a custom standard mix.

Table 3: Initial Calibration Standards Preparation, Low Level Soil

Standard Solution	Final Volume Reagent Water (ml)	Volume of Standard Added to Reagent Water (ul)					
		1ppb *	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Gas Mix (500ppm)	5	-	-	-		2.0	5.0
		-	-	-			
Mix 1 (combined) (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Mix 1 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-	-	-
Freon Mix							
AC (500ppm)	5	-	-	3.0	4.0	5.0	6.0
	50	10	20			-	-
Mix 2 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-		
Mix 3 (500ppm)	5					2.0	5
Propenes (50ppm)	-	-	-	-	-	-	-
	50	10.0	20.0		-	-	-
Propenes (Hi)(500ppm)	5	-	-	2.0	5.0	20	50
	-	-	-	-	-	-	-

[1]This level is also used as the Continuing Calibration Verification.

Table 3a: Initial Calibration Standards Preparation, Aqueous (LOW LEVEL)

Standard Solution	Volume of Standard Added to Reagent Water (ul)						
	0.5ppb*	1ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	0.5	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 3 (varied)	0.5	1	1	2	5	20	50
AC (500ppm)	2	4	4	4	10	20	40
1,4-Dioxane (500ppm)	15	30	-	-	-	-	-
Freons mix	0.5	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.5	0.5	0.5	1	2.5	10	25
Methanol Compensate	3000	2800	610	300	280	190	0
Final vol. (reagent water)	500ml	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 3b: Initial Calibration Standards Preparation, Aqueous

Standard Solution	Volume of Standard Added to Reagent Water (ul)					
	1.0ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	1	1	2	5	20	50
Mix 3 (varied)	1	1	2	5	20	50
AC (500ppm)	4	4	4	10	20	40
1,4-Dioxane (500ppm)	30	-	-	-	-	-
Freons Mix	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.25	0.5	1	2.5	10	25
Methanol Compensate	2800	610	300	280	190	0
Final vol. (reagent water)	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 4 : ICV Standard Preparation, Low Level Soil

Standard Solution	Concentration	Volume of Standard Added to 5.0 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	2	20
8260 SS (Separate lot)	50ppm (+varied)	2	20
AC SS (separate lot)	500ppm	3	300
Freon SS (Separate lot)	50ppm	2	20
Propenes SS(separate lot)	50ppm (varied)	2	20 (varied)

Table 4a: ICV Standard Preparation, Aqueous

Standard Solution	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	20	20
8260 SS (Separate lot)	5000ppm (varied)	20	20
AC SS (separate lot)	500ppm	4	400
Freons SS (Separate lot)	50ppm	20	20
Propenes (second source)	50ppm (varied)	20	20 (varied)

Table 5: SIM Initial Calibration Standards Preparation

Standard Solutions	Volume Standard Solution Added to Reagent Water (Final Concentration)							
8260 Mix 1 SIM (10ppm)	1 ul (0.02 ppb)	2 ul (0.04 ppb)	1 ul (0.05 ppb)	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1 .0 ppb)	10 ul (2.0 ppb)
1,4-Dioxane (500ppm)	4 ul (0.4 ppb)	2 ul (1.0 ppb)	1 ul (5.0 ppb)	1 ul (10 ppb)	1 ul (20 ppb)	2.5 ul (30 ppb)	5 ul (40 ppb)	10 ul (50 ppb)
SIM (ben/chl) 10ppm	1 ul (0.02 ppb)	1 ul (0.02 ppb)	1 ul (0.05 ppb	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1.0 ppb)	10 ul (2.0 ppb)
Final Vol. (reagent water)	**500ml**	**500ml**	**200ml**	**100ml**	**50ml**	**50ml**	**50ml**	**50ml**

levels 1 and 2 are respectively prepared in 500ml and 100ml final volumes
[1]**This level is also used as the Continuing Calibration Verification.**

Table 6 : SIM ICV Standard Preparation

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
SIM MIX1 SS (Second source)	10ppm	1	0.05
1,4-Dioxane SS	50ppm	20	5

TABLE 7
Characteristic Ions of Volatile Organic Compounds

Parameter	Primary ion	Secondary ion
1,1,1-Trichloroethane	97	99,117,119
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,1,2-Trichloroethane	97	83,85,99,132,134
1,1-Dichloroethane	63	65,83,85,98,100
1,1-Dichloroethene	96	61,98
1,1-Dichloropropene	75	110. 77
1,2,3-Trichlorobenzene	180	182
1,2,3-Trichloropropane	110	75
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromomethane	107	109
1,2-Dichloroethane	62	64,100,98
1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	65,114
1,2-Dichlorotrifluoroethene	67	117
1,2-Difluorotetrachloroethene	101	103, 167
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
1,4-Dioxane	88	58
1-Chloropropane	63	78
1-Methylnaphthalene	142	141
1-Propene	41	42
2,2-Dichloropropane	77	97

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
2,4,4-trimethyl-1-pentene	41	57, 97
2-Butanone	72	57
2-Chloroethyl vinyl ether	63	65, 106
2-Chloropropane	78	63
2-Chlorotoluene	91	126
2-Chloro-1,3-butadiene	88	53
2-Hexanone	43	58,100
2-Methylnaphthalene	142	141, 115
2-Nitropropane	39	42, 44
2-Octane	43	58
2-Octanol	45	55
4-Chlorotoluene	91	126
4-Methyl-2-Pentanone	43	58,100
Methacrylonitrile	67	41
Acetone	43	58
Acetonitrile	39	40, 41
Acrolein	56	55
Acrylonitrile	53	52
Allyl Alcohol	57	40, 39
Allyl Chloride	76	41
Amyl Acetate	43	70, 61
Benzene	78	--
Benzyl Chloride	91	126, 65
Bromobenzene	156	77, 158
Bromochloromethane	129	49, 130
Bromodichloromethane	83	85
Bromoform	173	171,175,
Bromomethane	94	96
Butyl Acetate	73	56, 43
Butyl Acrylate	73	56, 55
Butyl methacrylate	87	69
Camphene	93	121
Camphor	95	81
Carbon disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Chlortrifluoroethene	116	118
cis-1,3-Dichloropropene	75	77

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
Cyclohexane	56	84, 69
Cyclopentene	67	68, 68, 53
Dibromochloromethane	129	208,206
Dibromomethane	93	95, 174
Dichlorodifluoromethane	85	87
Dimethylnaphthalene (total)	141	156, 155
Epichlorohydrin	57	62, 49
Ethanol	46	45
Ethyl Acetate	70	61, 43
Ethyl Acrylate	55	56
Ethyl Ether	59	74, 75
Ethylbenzene	106	91,
Ethyl methacrylate	69	41, 99
Freon TF	101	103, 151, 85
Hexachlorobutadiene	225	223
Hexane	56	57, 86
Indan	117	118, 58
Iodomethane (methyl iodide)	142	127
Isobutyl Alcohol (Isobutanol)	43	41, 42
Isoprene	67	53, 59
Isopropanol	45	59
Isopropyl Acetate	43	61, 87
Isopropyl Ether (DIPE)	45	87
Isopropylbenzene	105	120
Methyl Acetate	43	74
Methyl Acrylate	55	85, 42
Methyl cyclohexane	83	55, 98
Methyl Methacrylate	100	69
Methyl tert-butyl ether (MTBE)	73	57
Methylene chloride	84	49,51,86
Methylnaphthalene (total)	142	141, 115
Naphthalene	128	--
n-Butanol	56	41, 43
n-Butylbenzene	91	92, 134
n-Heptane	57	43, 71
n-Pentane	72	57
N-Propanol	60	59
n-Propylbenzene	91	120
P-Isopropyltoluene`	119	134, 91
Propyl Acetate	43	61, 73
Propionitrile	54	52, 54

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
sec-Butylbenzene	105	134
Styrene	104	78,103
Tert-Amyl Methyl Ether	73	55, 87
Tert-butyl Alcohol	59	--
Tert-Butyl Ethyl Ether	59	87
Tert-Butylbenzene	119	91, 134
Tetrachloroethene	164	129,131,166
Tetrahydrofuran	42	72, 71
Toluene	92	91
Total Xylenes	106	91
trans,-1,3-Dichloropropene	75	77
Trans-1,4-dichloro-2-butene	53	75
Trichloroethene	130	95,97,132
Trichlorofluoromethane	101	103
Vinyl acetate	43	86
Dichlorofluoromethane	67	69
Chlorotrifluoroethene	116	118
1,2-tetrachlorodifluoroethane	101	103,167
1,2-Dichlorotrifluoroethane	67	117
Vinyl chloride	62	64
Isooctane	57	41, 56
1- Chlorohexane	91	93, 55, 56
1,2,4,5-Tetramethylbenzene	119	134, 91
4-EthylToluene	105	120, 77
Chlorotrifluoroethylene	66	116,118,85
Freon 114	85	87,135,137
t-Amyl Alcohol	59	55, 73, 43
1,4-Difluorobenzene	114	63
1,4-Diethylbenzene	119	105,134
Freon 123a	67	69, 117, 119
Butadiene	54	53, 39
4-Bromofluorobenzene (sur)	95	174,176
1,2-Dichloroethane-d4 (sur)	65	102, 104
Toluene-d8 (sur)	98	70,100
Fluorobenzene (istd)	96	77
Chlorobenzene-d5 (istd)	117	82,119
1,4-Dichlorobenzene-d4 (istd)	152	115,150